

N89-20307

CONTAINERLESS PROCESSING OF GLASS FORMING MELTS IN SPACE

by

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For presentation to:

Technical Review Committee
Microgravity Science Review
Marshall Space Flight Center

11 February 1987

ABSTRACT

The near weightlessness of a material in the reduced gravity environment of space offers the unique opportunity of melting and cooling glass forming compositions without a container. This reduces (or eliminates) the heterogeneous nucleation/crystallization which usually occurs at the walls of the container, thereby, extending the range of glass forming compositions. New glasses with interesting properties and glasses of ultra-high chemical purity can be made by containerless processing.

Based primarily on this idea, containerless glass forming experiments, which used a single axis acoustic levitator/furnace (SAAL), have been conducted on SPAR rocket flights, VI and VIII, and on Space Shuttle missions, STS-7 and STS-61A. The experiments on the Space Shuttle were designed to include other studies related to melt homogenization and mixing, development of techniques for preparing uncontaminated pre-flight samples, and simple shaping experiments. Knowledge about these factors is useful for planning and standardizing future containerless processing experiments in space.

Motion pictures of solid and liquid samples from the most recent experiment (STS-61A) showed that fluid melts can be successfully levitated and positioned with an acoustic field at temperatures up to 1550°C for several minutes. A ternary calcia-gallia-silica glass containerlessly melted in micro-g, provided evidence for a 2 to 3 fold increase in the tendency for glass formation in space. This is preliminary verification that heterogeneous nucleation is reduced in containerless melts. Melt homogenization for this calcia-gallia-silica melt was reasonably fast in space even with the lack of gravity-driven convection. Hot-pressing appears to be a feasible way of preparing precursor samples for use in containerless experiments in micro-g. Comparison of these results with those of earlier containerless experiments, from SPAR VI and STS-7, shows a progressive advancement in the science and technology of materials processing in space.

The objectives of future flight experiments on glass forming systems, which take advantage of the gravity-free environment of space, such as diffusion in the absence of gravity-driven convection, shaping of simple shapes using acoustic forces, preparation of glass composites from immiscible liquids, etc., are also briefly discussed.

1. INTRODUCTION

A serious factor that prevents many compositions from forming glass on earth is heterogeneous nucleation/crystallization which normally occurs at the melt-container interface. The impurities dissolved from the container material can also act as potential nucleation sites for heterogeneous crystallization as well as contaminating the melt. The net volume fraction, V_c/V , crystallized in a melt in time, t , is expressed as [1,2]

$$V_c/V = \pi/3 I(T)u^3(T)t^4 \quad (1)$$

where I and u are the nucleation and crystal growth rates, respectively, and both are functions of temperature, T . I in Eq. (1) generally includes both the homogeneous and heterogeneous nucleation rates. A foreign surface (or substrate) in contact with a melt provides heterogeneous nucleation sites which lower the thermodynamic barrier for nucleation. Thus, heterogeneous nucleation is more favorable than homogeneous nucleation. Regardless of the nucleation mechanism, crystallization of a melt can be avoided when it is cooled at a sufficiently high rate so that a glass is obtained, i.e., $V_c/V < 10^{-6}$. Depending on the characteristics of a particular system, there exists a minimum cooling rate for which the level of crystallization is just detectable ($V_c/V \approx 10^{-6}$). This rate is known as the critical cooling rate for glass formation, R_c .

I and u reach a maximum at some temperature where the increasing thermodynamic force for crystallization on cooling is counteracted by the increasing viscosity of the melt. The combined temperature dependence of I and u results in a nose-shaped curve on a temperature-time plot, see Fig. 1, which is known as temperature-time-transformation (or TTT) diagram [3].

The slope for the cooling curve which originates at the liquidus temperature and just touches the nose of the TTT diagram is considered to be R_c . The melt transforms to a crystalline or glassy solid when it is cooled at a rate $R < R_c$ (cooling curve intersects the TTT diagram) or $R > R_c$, respectively.

The near weightlessness of a material, solid or liquid, in the micro-g environment of space makes a container unnecessary. The absence of a container eliminates the possibility of a melt being contaminated by impurities from the container. Heterogeneous nucleation/crystallization can be suppressed, therefore, for containerless melts. The crystallization of such a melt will be determined primarily by the inner curve for homogeneous nucleation in Fig. 1. If homogeneous nucleation only occurs in space, then a melt can be cooled to glass at a cooling rate which is less than R_c on earth where heterogeneous nucleation is present because of the container. The suppression of heterogeneous nucleation in a containerless melt effectively lowers the nucleation rate, I , in Eq. (1) which decreases the volume fraction crystallized, V_c/V , or increases the glass formation tendency. This possibility for enhanced glass formation tendency for containerless melts in space can be

utilized to prepare glasses from compositions which may have interesting optical, electrical, or physical properties and whose crystallization during cooling cannot be avoided on earth. Furthermore, the elimination of impurities from the container makes it possible to prepare chemically ultrapure glasses which could be used for special applications such as lasers, optical waveguide fibers, etc. Preparing ultrapure glasses on earth can be a difficult to impossible task, especially if high melting temperatures are required and the melts are highly fluid and chemically reactive.

Based primarily on this idea, containerless glass melting experiments, which used a single axis acoustic levitator/furnace (SAAL), have been conducted in micro-g by R. A. Happe [4,5] of Rockwell International in SPAR rocket flights VI and VIII, and by ourselves on Space Shuttle missions, STS-7 [6] (June 1983) and STS-61A [7,8] (October 1985). The SAAL, which consists of a 4" x 4" x 4.5" furnace with four silicon carbide heating elements, a sound source, and a reflector for levitating and positioning a sample in the high temperature portion of the furnace, a mechanism for inserting and retrieving the sample from the furnace, and a motion picture camera for recording the sample while being processed in the SAAL, is described in detail elsewhere [9,10]. The SAAL used in rocket flights had the capability of processing only one sample, while that used in the Space Shuttle could process eight samples consecutively. Other subjects such as melt homogenization and mixing, developing techniques for preparing uncontaminated preflight (precursor) samples, and simple shaping experiments, were also investigated in the Space Shuttle experiments. The results from these experiments have provided valuable knowledge for the behavior of containerless melts at high temperatures and have been useful for planning and standardizing future containerless experiments in space.

The SPAR experiments have been included in this report since the final analysis of the SPAR VI sample was our responsibility [9]. Also, the same SAAL design was used for all the containerless experiments, so a brief review of the SPAR experiments provides a better chronological description of the operational performance of the SAAL for processing multicomponent, glass forming melts in space. However, the present paper primarily deals with the results of the most recent experiment conducted on the Space Shuttle STS-61A which used the MEA/A-2 (Materials Experiment Assembly) flight hardware. The earlier experiments will be referred to and discussed only when a comparison of the results is needed.

Finally, the objectives of future flight experiments on glass forming systems, which take advantage of the reduced gravity environment of space, such as diffusion in the absence of gravity-driven convection, shaping of simple shapes using acoustic forces, and the study of immiscible liquids, are briefly discussed.

2. EXPERIMENTAL

Eight spherical samples, $\approx 6 \pm 1$ mm in diameter, were used in the MEA/A-2 experiment, STS-61A. Each sample had one or more of the following objectives: 1) to obtain quantitative evidence for the suppression of heterogeneous nucleation/crystallization (or enhanced glass formation) in containerless melts in micro-g, 2) to study melt homogenization in the absence of gravity-driven convection, 3) to perform comparative property analysis of glasses melted on

earth and in micro-g, 4) to develop procedures for preparing precursor samples that yield bubble free, high purity, chemically homogeneous melts in micro-g, 5) to determine the feasibility of shaping glass shells in micro-g for use as laser fusion targets, and 6) to assess the operational performance of the SAAL for processing multicomponent, glass forming melts in micro-g. The composition, purpose, and the temperature/time planned for the containerless processing of each sample are given elsewhere [7,8].

Each sample was held in an individual sample injector/retrieval system, which was fitted in a carousel assembly in the SAAL. The sample processing procedure was fully automated. During operation, the sample injector moved inside the furnace through a gate and released the sample for stable positioning at the potential energy minimum of the sound field confined between the sound source and reflector of the SAAL. After melting, the sample was cooled by temporarily turning off the power to the furnace and opening a gate on one side of the furnace wall. At the completion of processing, the sample was retrieved, the carousel assembly rotated, and the second sample automatically moved to the position of the sample injection gate.

The critical cooling rate for glass formation, R_c , was used as the quantitative parameter for the suppression of heterogeneous nucleation/crystallization. As discussed earlier, if heterogeneous nucleation caused by a container is absent in containerless melts, then it should be possible to quench such melts to glass using cooling rates which are less than R_c measured on earth where heterogeneous nucleation is present. Thus, the ratio R_c^e (earth) to R_c^s (space) for melts quenched to glass in micro-g should exceed unity and the numerical value of this ratio can be used as a quantitative measure of the degree to which glass formation is enhanced, or, conversely heterogeneous nucleation/crystallization is suppressed in containerless melts. Samples whose R_c on earth is several times higher than the cooling rate of the SAAL furnace used in micro-g ($\approx 5^\circ\text{C/s}$) was used to provide evidence for enhanced glass formation in micro-g.

Melt homogenization in the absence of gravity-driven convection in micro-g was to be investigated by observing the level of homogeneity achieved in precursor samples made with known chemical inhomogeneities. Hot-pressed samples containing relatively large SiO_2 particles (100 - 300 μm) and a sample which contained a colored spot on its external surface were used for this purpose. In addition, a sodium-borate sample containing gas bubbles at known locations was used to provide information for the behavior of gas bubbles in liquid melts and supplemental data for melt homogenization in micro-g.

Glass shells of uniform sphericity and wall thickness, and several millimeters in diameter have important uses in laser fusion technology as inertial confinement fusion (ICF) targets. They are essentially impossible to fabricate on earth, however, due to gravity induced distortion. As there is no buoyant force in micro-g, a bubble initially present inside a glass should not necessarily escape when the glass is remelted in space. On the other hand, surface tension forces should reshape the sample into a spherical shell, perhaps of uniform wall thickness, when it is levitated and melted without a container. A soda-lime-silica glass shell containing an air bubble ≈ 3.75 mm in diameter was used for this purpose. Lastly, several physical, optical, thermal, and mechanical properties for glasses made in micro-g were compared with the same properties measured for glasses of identical composition made on earth.

An important practical objective of this experiment was to determine the suitability of hot-pressed, precursor samples for use in containerless melting. Hot-pressed precursor samples have the advantage of being easily prepared without contamination from a container. A sample larger than that needed can be hot-pressed, with only the uncontaminated core being used for the experiment in space. It is important to know the maximum size of the chemical inhomogeneities that can be tolerated in a hot-pressed precursor which will yield a chemically homogeneous, multicomponent melt in a reasonable time when melted in micro-g.

3. RESULTS AND DISCUSSION

The film and flight data for this experiment indicate that the first three samples were processed in a containerless manner according to the planned time-temperature schedule. None of the remaining five samples were inserted into the furnace, levitated, or melted. Of the three samples processed, sample 2 which was a hot-pressed $35.7\text{CaO}-39.3\text{Ga}_2\text{O}_3-25\text{SiO}_2$, mol%, composition had more than one scientific objective on MEA/A-2, i.e., 1) to verify enhanced glass formation in micro-g, 2) to evaluate melt homogenization and mixing in the absence of gravity-driven convection, and 3) to assess precursor preparation technique. The planned and observed temperature-time profile for the containerless melting of sample 2 is given in Fig. 2. The first sample was a non-melting alumina and was used for an engineering check-out of the SAAL.

A. Flight Events for Sample 2

When sample 2 was recovered from the MEA/A-2 flight hardware, it was stuck to one side of the platinum wire cage, see Fig. 3, which surrounded the sample primarily for the purpose of preventing it from being lost if, for any reason, it should escape from the acoustic energy well. The motion picture film for sample 2 clearly showed that the sample struck and adhered to the cage when the cooling gate of the SAAL opened for cooling. The instability of sample 2 and its movement out of the acoustic well coincided with the opening of the cooling gate. Several factors may have caused sample 2 to escape from the acoustic well. Opening the cooling gate could have produced a temperature gradient, which in turn may have produced pressure gradients inside the furnace which were sufficient to displace the sample from the acoustic energy well.

B. Enhanced Glass Formation for Sample 2

When examined with a scanning electron microscope (SEM), that part of sample 2 touching the cage wires was seen to be crystalline. The crystallized region extended about 1 mm along the surface of the sample from both sides of the wire and slightly less than 1 mm (0.8 to 0.9 mm) into the interior of the sample. The central part of sample 2 which did not touch the wires was glassy. A few scattered crystals confined only to the external surface of the glassy region was also observed. The sample is estimated to contain 70 to 80% glass, the remainder being crystalline. It is believed that sample 2 would have been totally glassy if it had not touched the cage which produced heterogeneous nucleation/crystallization. The average cooling rate between 1450 and 1000°C, as calculated from two thermocouples within about 1 cm of the sample on MEA/A-2, was $\approx 4.2^\circ\text{C/s}$. This is lower than the critical cooling

rate, R_c , measured on earth ($11.3 \pm 2^\circ\text{C/s}$) for a sample of identical composition by a factor of 2.7. This indicates that containerless melting in space has improved the glass formation tendency of this calcia-gallia-silica composition by a factor of between 2 and 3. Based on the measured R_c value and nose temperature ($\approx 1215^\circ\text{C}$), a TTT diagram for this composition having a sample size identical to sample 2 (diameter ≈ 6.5 mm) has been estimated and is shown in Fig. 4. Superimposed on the TTT diagram is the actual cooling curve of flight sample 2, which is seen to pass through the crystallized region inside the TTT diagram. This indicates that a sample cooled on earth at a rate equal to that used in the flight experiment would be totally crystalline. The fact that sample 2 returned as a glass clearly demonstrates that containerless melting increased the glass formation tendency of this calcia-gallia-silica composition. Although the data are limited, the most likely explanation for these results is the reduction in heterogeneous nucleation in the containerless melted sample.

C. Melt Homogenization for Sample 2

As mentioned earlier, the sample 2 precursor was made by hot-pressing crystalline powders. This sample contained random heterogeneities in the form of 100 - 300 μm silica particles dispersed in the fine-grained calcia-gallia matrix, see Fig. 5. No remnants of these silica particles or regions rich in silica were detectable by SEM on either the external surface or in the bulk of sample 2 after melting in space. The relative composition at different locations on the external surface and in the interior (fracture surface) of sample 2 was determined by energy dispersive x-ray analysis (EDAX) and is shown graphically in Fig. 6. The compositions determined at different, randomly chosen locations in the bulk are close to the actual composition and the scatter in composition is significantly less than that for the external surface. This shows that the bulk of sample 2 is more homogeneous than the external surface which is also consistent with the fact that the external surface was partially crystallized. The complete dissolution of the large silica particles to form a reasonably homogeneous glass in a melting time of ≈ 4 min at an average temperature of 1475°C suggests that the chemical homogenization process is reasonably fast in space for this low viscosity melt, even in the absence of gravity-driven convection. Hot-pressing appears to be a feasible way of preparing high purity, precursor samples for use in flight experiments which will yield a chemically pure, homogeneous glass in micro-g within a reasonable time.

D. Comparison of Properties for the Glasses Melted in Space (Sample 2) and on Earth

Due to its small size and partial devitrification, only a few properties could be measured for flight sample 2. The refractive index, dispersion, and Abbe number for this glass, as measured by the Becke line technique, are compared in Table I with those of a glass of identical composition melted on earth. While there are slight differences in the properties of the two glasses, the differences are not considered to have practical significance. The crystallization of the space and earth-melted glasses were also studied by differential thermal analysis (DTA) at a heating rate of 10°C/min . A comparison of the DTA curves in Fig. 7 shows that the crystallization for the space and earth melted glasses is essentially identical. The appearance of a large exothermic peak with a small shoulder on the low temperature side in the DTA curve shows that two compounds crystallize from this glass. They have been

Table I. Refractive index, dispersion, and Abbe number for the 35.7CaO-39.3Ga₂O₃-25SiO₂, mol%, glass melted in space and on earth.

	Space Sample 2 (MEA/A-2)	Earth sample
Refractive Index (± 0.001)	1.729	1.712
Dispersion	0.028	0.018
Abbe Number (± 3.5)	26.1	40.5

identified by powder x-ray diffraction (XRD) as β -Ga₂O₃ and (2CaO·Ga₂O₃·SiO₂). The same two compounds were also observed when the crystallized region of the space melted sample was examined by SEM and EDAX, see Fig. 8, and by Debye Scherrer XRD. The larger peak at $\approx 887^\circ\text{C}$ in Fig. 7 corresponds to the crystallization of (2CaO·Ga₂O₃·SiO₂) as determined by examining the crystallization of a glass whose composition is (2CaO·Ga₂O₃·SiO₂). The smaller peak ($\approx 855^\circ\text{C}$) in Fig. 7 is attributed to the crystallization of β -Ga₂O₃.

The infrared transmission of the space and earth melted calcia-gallia-silica glass are compared in Fig. 9. There is no significant difference in the IR transmission of these two glasses.

E. Analysis of Sample 3

Sample 3, which was a soda-lime-silica glass shell containing a large air bubble, was used to assess the feasibility of removing the irregularities from its inner surface and reshaping it into a spherical shell of uniform wall thickness by remelting it in micro-g. Radiographic analysis of the post flight sample confirmed that it was no longer a shell, but an ellipsoidal piece of solid glass whose largest and smallest diameter differed by ≈ 0.521 mm (diameter, max = 5.334, min = 4.813 mm). The reason why the bubble escaped and how the sample deformed to an ellipsoidal shape is not clear at this point, and is still being investigated. One possible explanation for the escape of the bubble is the existence of a temperature gradient, which could establish a surface tension gradient in the sample. Marangoni flow could have caused the bubble to move to the region of lower surface tension (hot spot) and escape from the sample.

The possibility that sample 3 could have been distorted by rapid spinning when it was molten has been examined. Calculations show that a rotational speed of ≈ 1220 rpm would be required to produce the shape and dimensions identical to flight sample 3 [11]. There is no definite evidence showing whether sample 3 was spinning while molten, but this speed is considered unrealistically high. Another possibility is that the sample was deformed by acoustic forces since recent calculations indicate [12] that these forces increase during cooling and may not be negligible as has been assumed previously.

F. Operational Performance of SAAL

The operation of the SAAL in space for high temperature liquid samples was generally satisfactory. All the principal features of the SAAL such as sample insertion and levitation, temperature control of the furnace, etc., operated as intended for the first three samples. An excellent photographic record of the sample processing sequences in micro-g, which gives a good optical view of the samples and their movements at elevated temperature, was obtained. The reason why the furnace did not operate beyond the third sample is not precisely known, but it appears that a lack of liquid coolant for the flight hardware created an overtemperature condition which caused the premature shutdown of the furnace. However, the performance of the SAAL in the MEA/A-2, STS-61A experiment is considered to be significantly improved compared to that for the MEA/A-1, STS-7 experiment [6].

This is the first time that liquid samples have been successfully levitated in space at high temperatures (1250 to 1500°C) for periods of 4 to 12 min. This time period is reasonably long compared to that for the earlier SPAR VI, SPAR VIII, and STS-7 containerless experiments. The glass forming melt was levitated only for 27 s in SPAR VI [4,9] and for 83 s in SPAR VIII [5] before it touched and stuck to the platinum wire cage. The only sample which showed evidence of being partially melted in MEA/A-1 [5] had a composition identical to sample 2 in MEA/A-2, but it is difficult to say how long it was levitated in the SAAL as no photographic evidence was obtained. This sample was also found stuck to the cage wires when removed from the MEA/A-1 flight hardware and was totally crystalline.

The sample injection mechanism worked perfectly for MEA/A-2 and the sample was more stable compared to the earlier experiments. In SPAR VI and VIII, the sample moved about and touched the cage several times just after being released inside the platinum cage in the SAAL furnace. The samples then remained stationary for a brief period (27 s in SPAR VI and 83 s in SPAR VIII) whereupon they again started moving and stuck to the cage long before the cooling gate was opened for cooling. This indicates that the displacement of these samples out of the acoustic well was caused not by any external force but by an instability in the acoustic field itself. No such instability of the acoustic field was observed for any of the three samples processed in MEA/A-2, except that sample 2 escaped from the acoustic well when the cooling gate was opened.

4. RATIONALE FOR FUTURE EXPERIMENTS IN MICROGRAVITY

In future containerless experiments on glass forming systems, it is planned to study other phenomena requiring a gravity-free environment. For example, mass transport by diffusion is nearly always masked by that due to gravity-driven convection on earth. In low gravity, diffusion-controlled transport can be studied independently and more accurate information about the atomistic or microscopic nature of a material can be obtained.

It is difficult and often impossible to independently study homogeneous and heterogeneous nucleation in fluid melts on earth. This can be accomplished in micro-g for containerless melts and basic information about the nucleation mechanism can be obtained.

In fluid liquids, such as alkaline earth borates and metals, it is difficult to study the mechanism and kinetics of phase separation (immiscible liquids) on earth, because the two liquids have different densities and separate too rapidly for measurements. In space, where density-driven motion is greatly suppressed, phase separation in liquids can be studied without interference from gravity-driven convection. Studies in space should provide valuable scientific information for these interesting systems and offer the potential of yielding composite materials with interesting microstructures and properties. The measurement of surface properties (surface tension, emissivity, vaporization) of a solid or liquid material is another important area of investigation. To obtain uncontaminated surfaces on earth is a difficult to impossible task, but containerless processing in micro-g offers the opportunity to investigate an uncontaminated and unsupported surface. Many properties such as strength, surface flow, surface nucleation and crystallization, ion-exchange reaction, surface-surface interaction, and rate of gas evolution or vaporization, can be studied to gain a better understanding of the physical, chemical, and structural characteristics of surfaces.

Forces which are weak compared to gravity-driven convection, but may become significantly large in the absence of convection, can also be studied in micro-g. The effect of these forces on the processing of a material can be investigated. For example, surface tension which is expected to be important in micro-g can be used to homogenize a melt by Marangoni flow. Forces arising from electric, magnetic, or acoustic fields can be used to control the shape of a melt in micro-g, thereby, avoiding the mechanical stress or distortion normally introduced in the material as a result of treatments like cutting, grinding, or polishing.

5. CONCLUSIONS

The following conclusions summarized below are based on all flight experiments conducted to date, but especially on the results of the MEA/A-2 flight experiment.

A. Enhanced Glass Formation

Two glass forming compositions (calcium-gallia-silica and soda-lime-silica) were successfully melted and cooled to glass while levitated in space. The calcium-gallia-silica composition requires a cooling rate ≈ 11 to 12°C/s to form glass on earth for the sample size used in the space experiment. This cooling rate is 2 to 3 times higher than the cooling rate ($\approx 4^\circ\text{C/s}$) for the MEA/A-2 experiment which yielded a glass for this composition. Clearly, the glass formation tendency for this calcium-gallia-silica composition is increased by 2 to 3 times when melted in space without a container.

B. Melt Homogenization and Precursor Preparation Technique

A usable and homogeneous glass was obtained for the calcium-gallia-silica composition for the melting time and temperature used in the flight experiment. Initially, this sample was made by hot-pressing crystalline powders and contained random heterogeneities of pure silica particles. Chemical homogenization of this low viscosity melt was reasonably fast even in the absence of gravity-driven convection. Hot-pressing appears to be a feasible way to prepare chemically pure samples for materials processing in space.

C. Comparison of Properties

The comparison of selected properties for the space and earth-melted calcia-gallia-silica glasses shows that the refractive index, dispersion, and Abbe number are slightly different, but there is no detectable difference in their infrared transmission or crystallization behavior. For this particular glass composition, melting in space produced no important difference in properties.

D. Shaping

Since the bubble escaped from the soda-lime-silica glass shell when it was remelted in space, the feasibility of shaping a glass shell in micro-g has not been fully demonstrated. The reshaping of glass shells in space is still considered feasible once the reason responsible for bubble motion is identified.

E. Operational Assessment of SAAL

The operation of the single axis acoustic levitator/furnace in space for high temperature liquid samples was generally satisfactory for the MEA/A-2 experiment. For the first time, liquid samples have been successfully levitated in space at high temperatures (1250 to 1500°C) for periods of 4 to 12 minutes. All the principal features of the SAAL such as sample insertion and levitation, temperature control of the furnace, etc. operated as intended for the first three samples. An excellent photographic record of the sample processing sequences in micro-g, which gives a good optical view of the samples and their movements at elevated temperature, was also obtained.

6. FUTURE WORK

Based on the operational performance of the SAAL and the results obtained from the glass melting experiments, it is believed that a valuable advancement in the science and technology of materials processing in space has been achieved. Nevertheless, the basic scientific objectives regarding enhanced glass formation, melt homogenization and mixing, bubble behavior, and reshaping simple shapes in space have not been completely fulfilled. The results of the MEA/A-2 experiment which demonstrate the enhancement of glass formation in space are encouraging and need to be verified for other potentially useful compositions.

ACKNOWLEDGEMENT

Thanks are due to Dr. N. J. Kreidl, consultant on this research, for many helpful discussions and suggestions, to Mr. F. Gac of Los Alamos National Laboratory for supplying the soda-lime-silica glass shell, to Dr. C. Rey of Intersonics, Inc. for many practical suggestions, and to Dr. R. S. Subramanian of Clarkson University for providing the sodium-borate sample.

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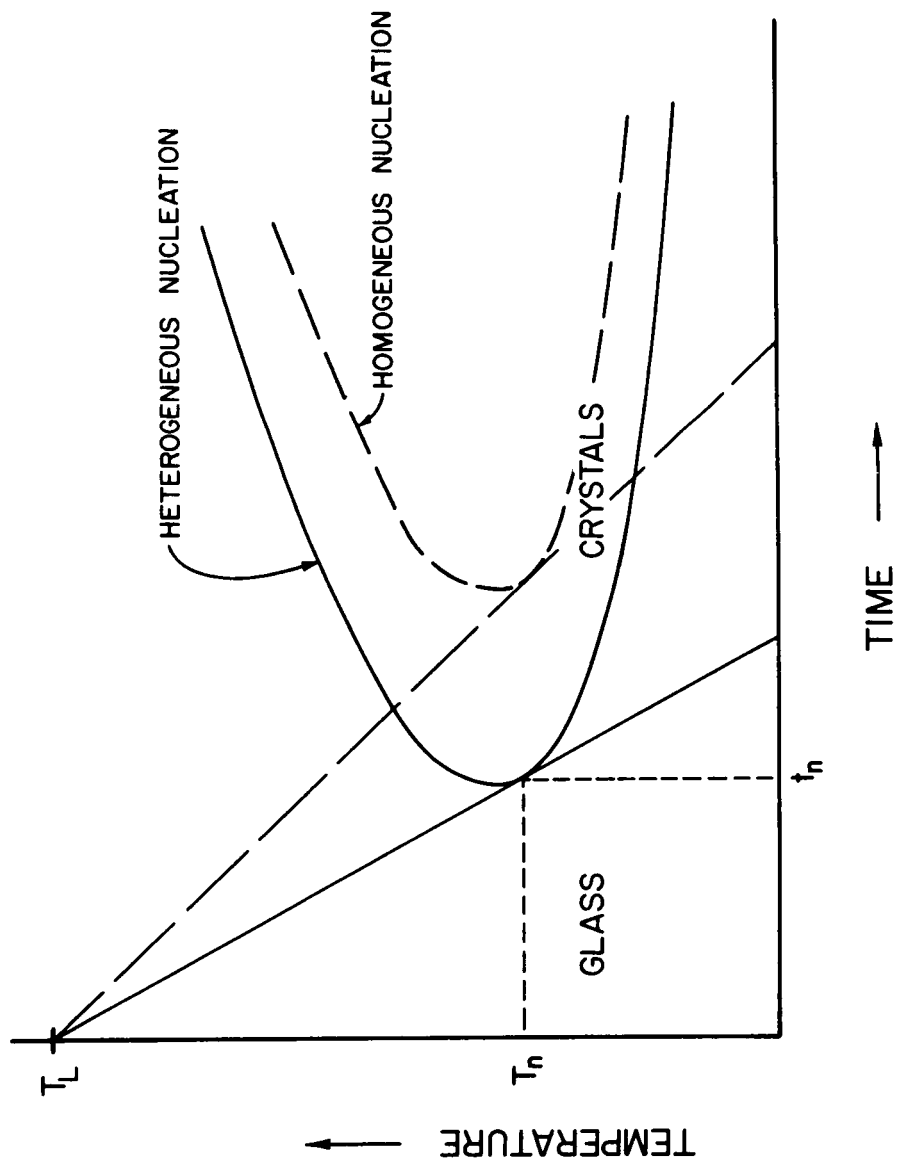


Figure 1. Typical time-temperature-transformation (TTT) diagram for a glass forming material.

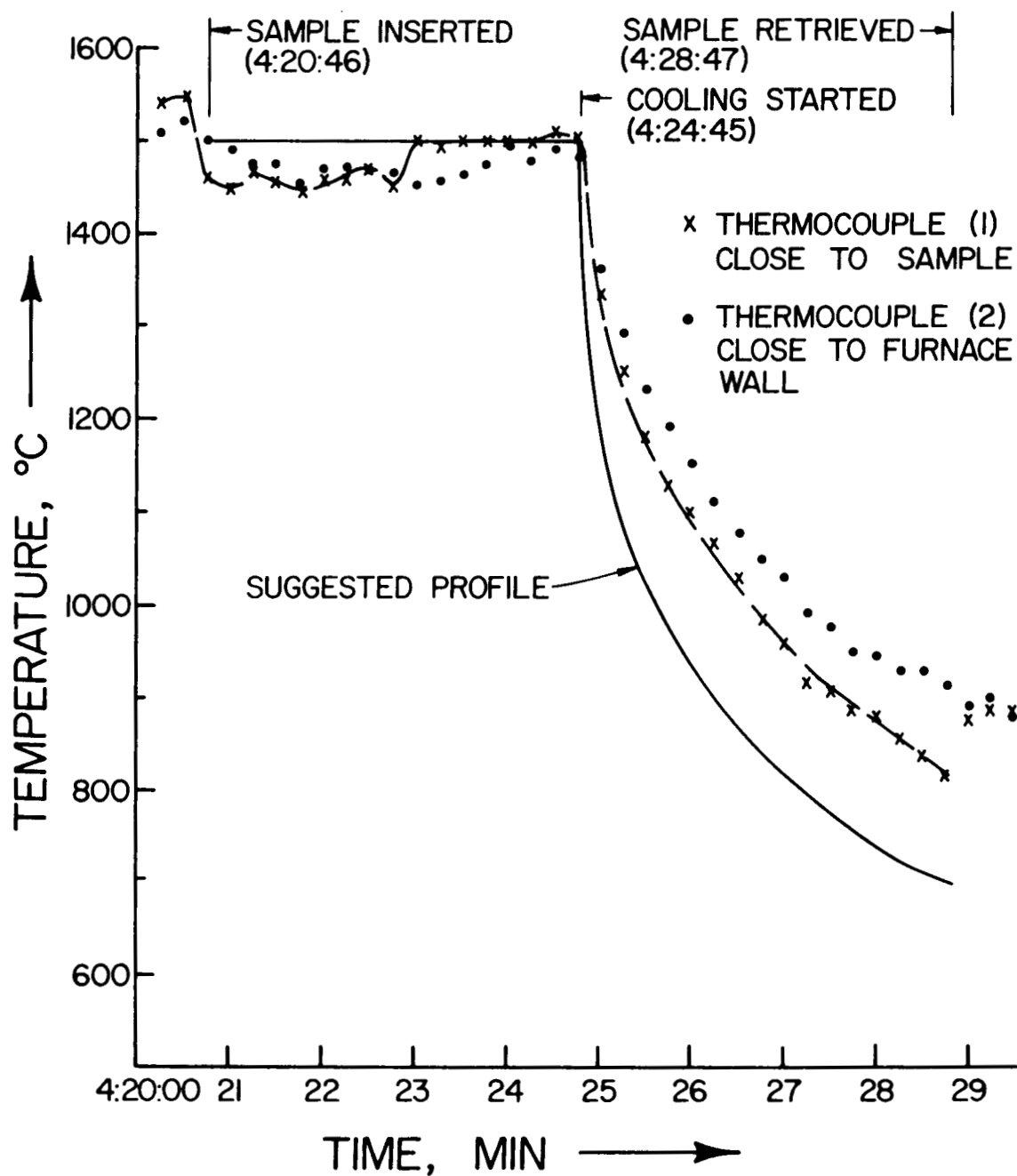


Figure 2. The planned and observed time-temperature profile for 35.7CaO-39.3Ga₂O₃-25SiO₂, mol%, hot-pressed sample (#2) when processed in the single axis acoustic levitator, MEA/A-2.

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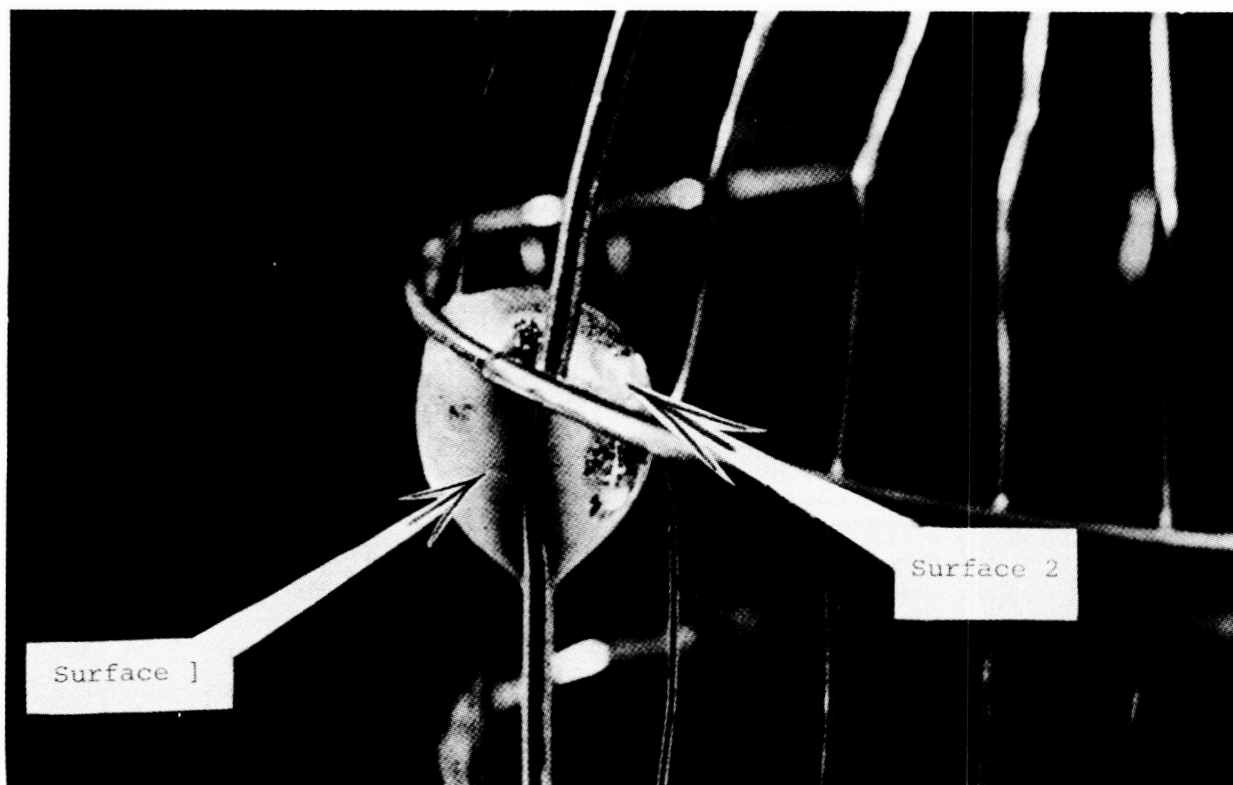


Figure 3. Flight sample 2 ($35.7\text{CaO}-39.3\text{Ga}_2\text{O}_3-25\text{SiO}_2$, mol%, hot-pressed) stuck to platinum wire cage while levitated and melted in space (MEA/A-2).

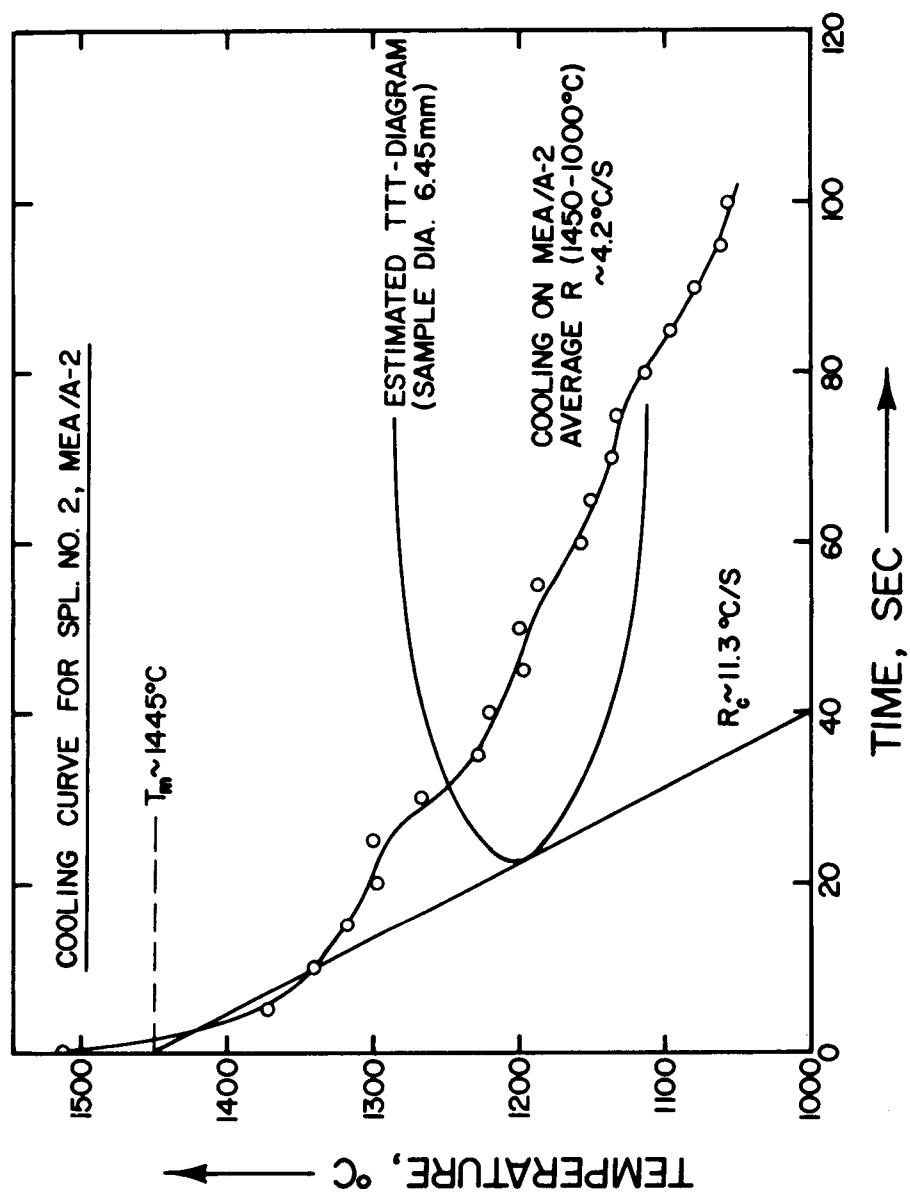


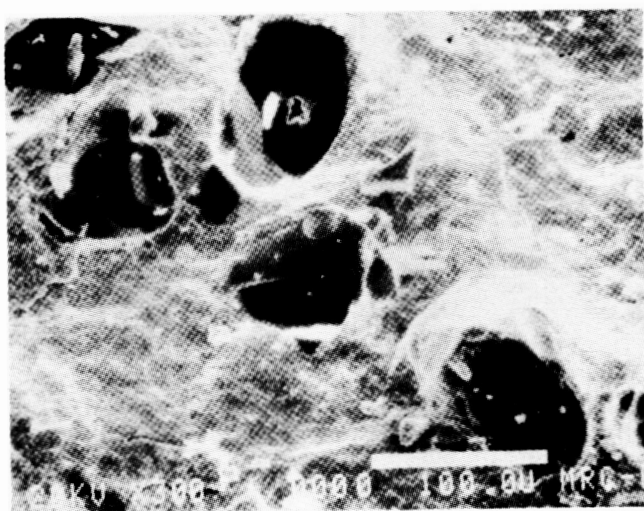
Figure 4. The estimated TTT diagram for the 35.7CaO-39.3Ga₂O₃-25SiO₂, mol%, composition for sample diameter ≈ 6.5 mm and the cooling rate measured for sample 2 in MEA/A-2.



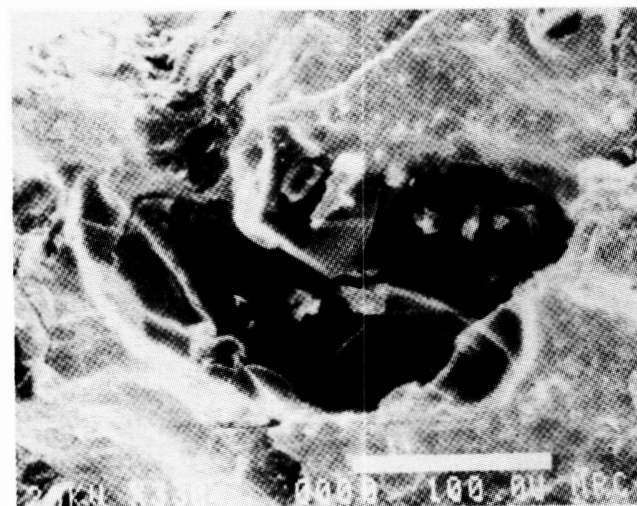
A



B



C



D

Figure 5. Appearance of the interior of the hot-pressed $35.7\text{CaO}-39.3\text{Ga}_2\text{O}_3-25\text{SiO}_2$, mol%, precursor as seen by SEM. Dark regions are silica particles. B is the back scattered photograph of the sample shown in A. C shows silica particles ≈ 100 micrometer size while larger silica particles ≈ 300 micrometer in size are shown in D.

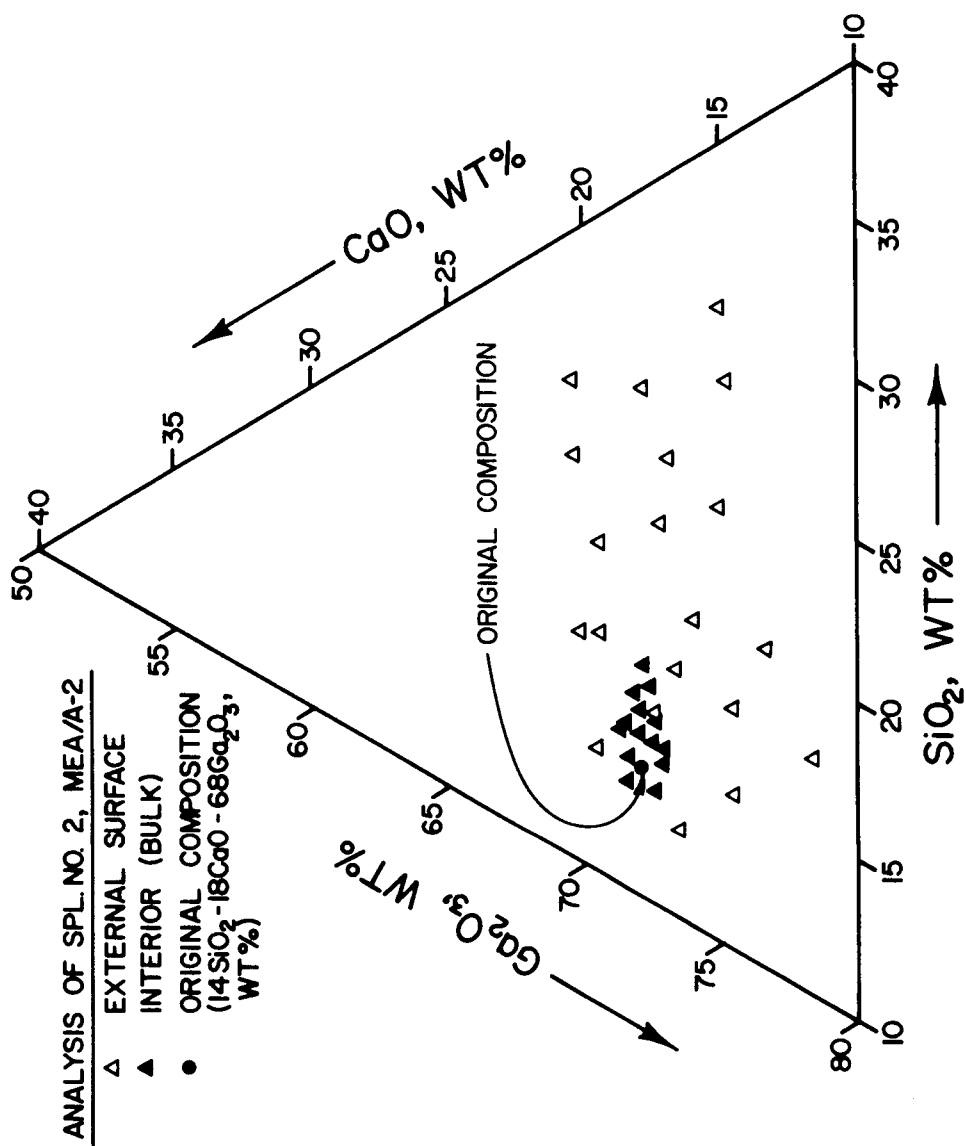


Figure 6. Composition of the external surface and the interior of flight sample 2 (35.7CaO-39.3Ga₂O₃-25SiO₂, mol%, hot-pressed) as determined by EDAX.

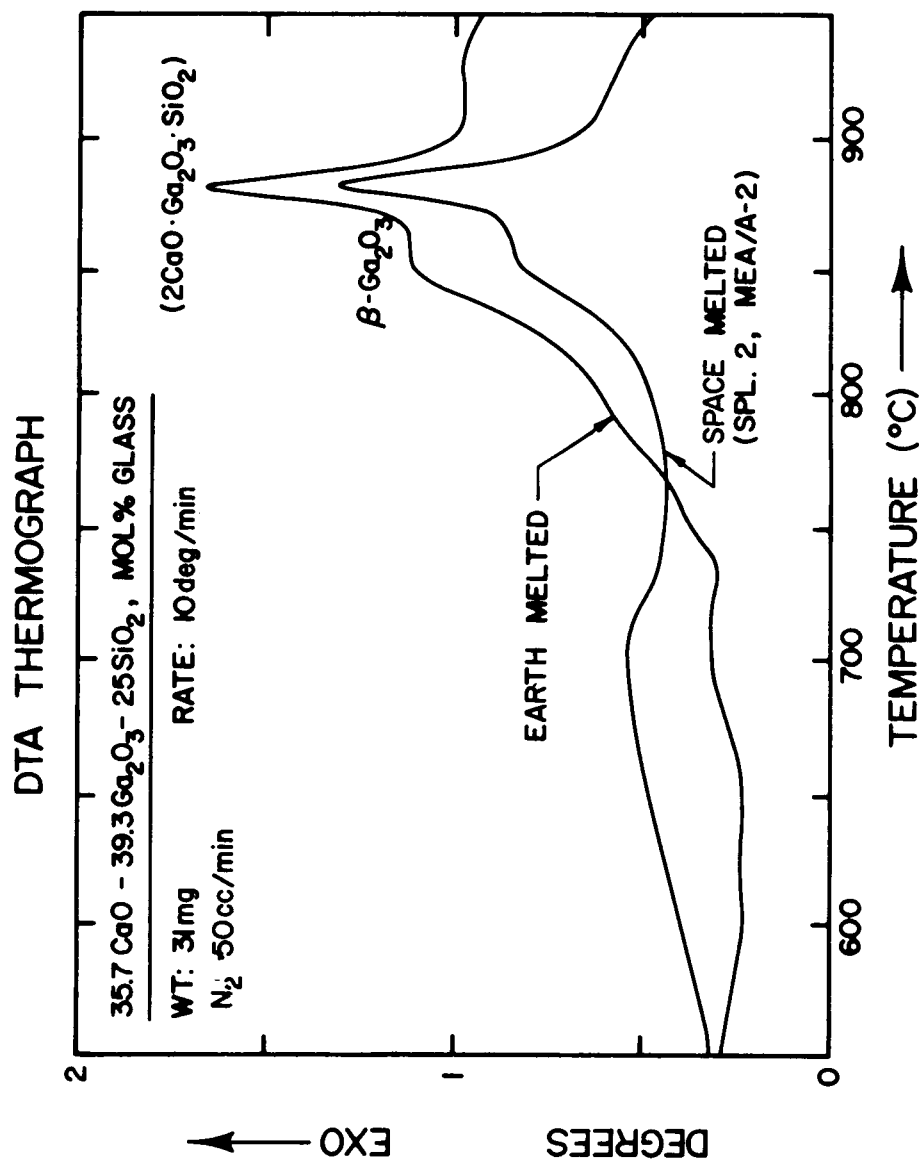
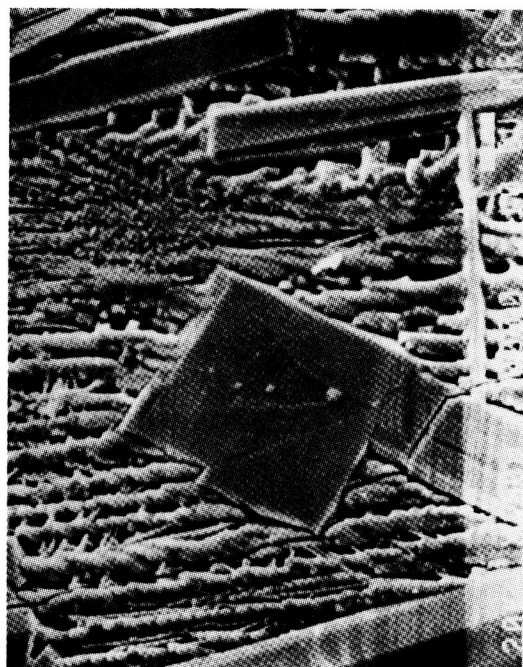
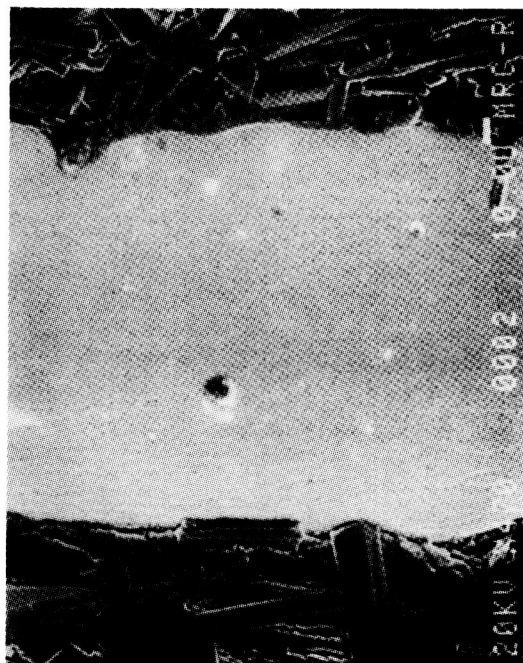


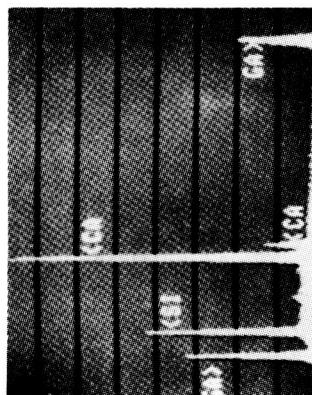
Figure 7. DTA thermogram for the 35.7CaO-39.3Ga₂O₃-25SiO₂, mol%, glasses melted in space (MEA/A-2) and on earth.



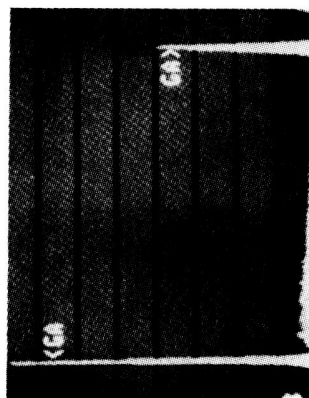
B



A



Y



X

Figure 8. External surface of flight sample 2 ($35.7\text{CaO}-39.3\text{Ga}_2\text{O}_3-25\text{SiO}_2$, mol%, hot-pressed) in the vicinity of the vertical platinum wire imbedded in the sample (shown as surface 1 in Fig. 3). The wide light band in A is the platinum wire imbedded in the sample. Crystals are visible on both sides of the platinum wire. Two crystalline phases, as shown in B, were observed. The plane flat crystals contain Ga only (see X). The rough needle shaped crystals contain Ca, Ga, and Si (see Y).

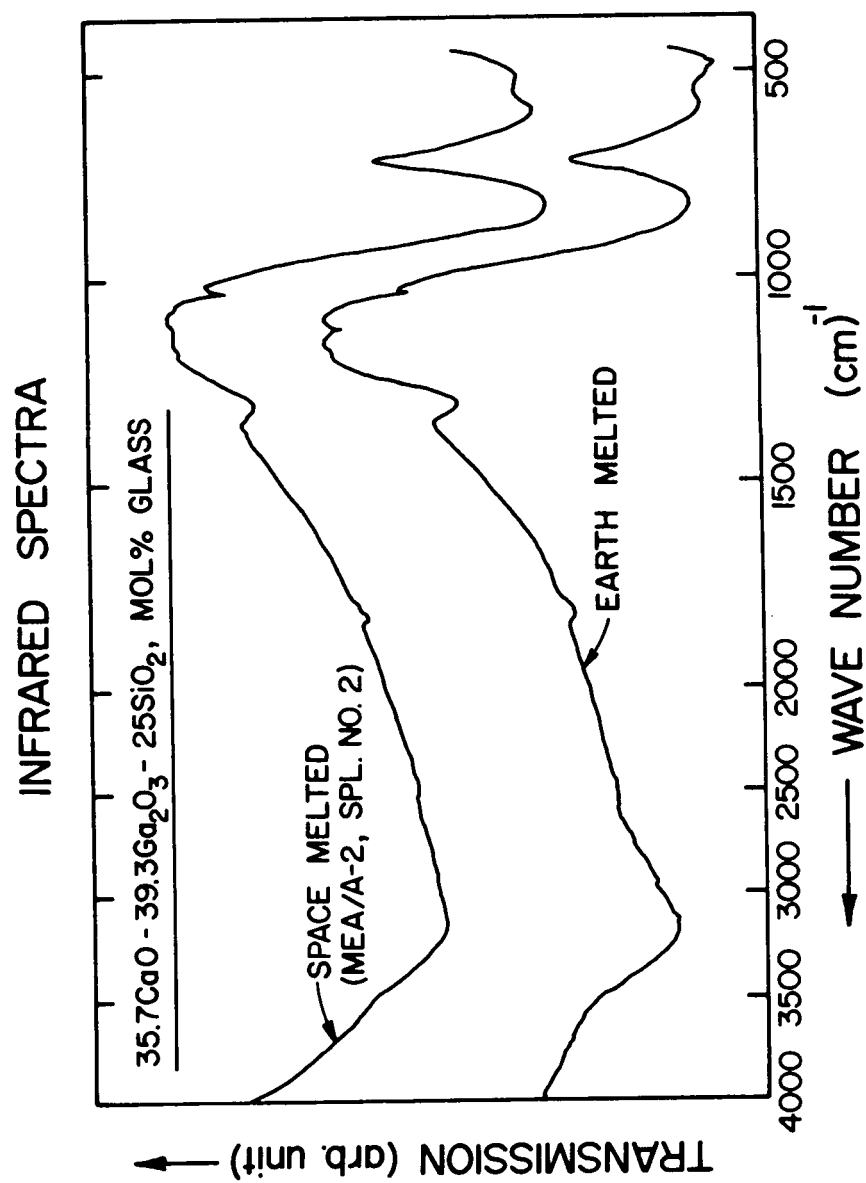


Figure 9. IR transmission for the 35.7CaO-39.3Ga₂O₃-25SiO₂, mol%, glasses melted in space (MEA/A-2) and on earth.